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METHOD OF CONTROLLING WEEDS

The present invention relates to a method of providing season-long control of unwanted vegetation from a single treatment.

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The protection of crops from weeds and other vegetation which inhibit crop growth is a constantly recurring problem in agriculture. To help combat this problem, researchers in the field of synthetic chemistry have produced an extensive variety of chemicals and chemical formulations effective in the control of such unwanted growth. Chemical herbicides of many types have been disclosed in the literature and a large number are in commercial use.

Agricultural pesticide manufacturers have identified the need for broad-spectrum, long-acting pesticidal products. Single active ingredient formulations rarely meet such requirements, and thus combination products, perhaps containing up to four complementary biologically active ingredients, need to be developed. Many combination products (i.e. products containing more than one active ingredient) are currently available, but none of these provide broad-spectrum, season-long control from a single application of the product.

It is therefore an object of the present invention to provide a method of providing season-long control of unwanted vegetation from a single treatment.

The individual compounds used in the method of the invention are independently known in the art for their effect on plant growth. For example, the herbicidal cyclohexanedione compound 2-(2'-nitro-4'-methylsulphonylbenzoyl)-1,3-cyclohexanedione (mesotrione) is a selective herbicide disclosed in U.S. Patent No. 5,049,046 along with a number of other cyclohexanedione compounds. The acetamides, in particular 2-chloro-N-(2-ethyl-6-methylphenyl)-N-(2-methoxy-1-methylethyl)acetamide (metolachlor), are a known class of compounds with herbicidal activity. Glyphosate is a non-selective herbicide available *inter alia* under the tradename Touchdown[®]. Although all these components are herbicidally active on their own when applied to unwanted vegetation, none of them individually are able to provide the broad-spectrum season-long control from a single application that is now being required by the agricultural industry.

Accordingly, the present invention provides a method for the season-long control of unwanted vegetation, said method comprising a single application of a herbicidal combination comprising a 2-(substituted benzoyl)-1,3-cyclohexanedione or metal chelate

thereof, glyphosate or a salt thereof and an acetamide to the locus of said unwanted vegetation. By the term 'season-long control' we mean that only one application of the herbicidal composition is required per season for any given crop, and that the unwanted vegetation will remain under control for the duration of the season. The 'season' is generally up to 120 days long, for example from 40 to 120 days, such as 50 to 120 days long.

A second aspect of the invention provides the use of a herbicidal combination comprising a 2-(substituted benzoyl)-1,3-cyclohexanedione or metal chelate thereof, glyphosate or a salt thereof and an acetamide, for the season-long control of unwanted vegetation, by a single application of the combination.

Suitably, the 2-(substituted benzoyl)-1,3-cyclohexanedione for use in the present invention is a compound of formula (I)

$$(Q)_p \xrightarrow{Q} (Z)_n \qquad (I)$$

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wherein X represents a halogen atom; a straight- or branched-chain alkyl or alkoxy group containing up to six carbon atoms which is optionally substituted by one or more groups -OR¹ or one or more halogen atoms; or a group selected from nitro, cyano, -CO₂R², -S(O)_mR¹, -O(CH₂)_rOR¹, -COR², -NR²R³, -SO₂NR²R³, -CONR²R³, -CSNR²R³ and -OSO₂R⁴;

R¹ represents a straight- or branched-chain alkyl group containing up to six carbon atoms which is optionally substituted by one or more halogen atoms;

R² and R³ each independently represents a hydrogen atom; or a straight- or branched-chain alkyl group containing up to six carbon atoms which is optionally substituted by one or more halogen atoms;

R⁴ represents a straight-or branched-chain alkyl, alkenyl or alkynyl group containing up to six carbon atoms optionally substituted by one or more halogen atoms; or a cycloalkyl group containing from three to six carbon atoms;

each Z independently represents halo, nitro, cyano, $S(O)_m R^5$, $OS(O)_m R^5$, C_{1-6} alkyl, C_{1-6} alkoxy, C_{1-6} haloalkyl, C_{1-6} haloalkoxy, carboxy, C_{1-6} alkylcarbonyloxy, C_{1-6} alkylcarbonyl, amino, C_{1-6} alkylamino, C_{1-6} dialkylamino having independently the stated number of carbon atoms in each alkyl group, C_{1-6}

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alkylcarbonylamino, C_{1-6} alkoxycarbonylamino, C_{1-6} alkylaminocarbonylamino, C_{1-6} dialkylaminocarbonylamino having independently the stated number of carbon atoms in each alkyl group, C_{1-6} alkoxycarbonyloxy, C_{1-6} alkylaminocarbonyloxy, C_{1-6} dialkylcarbonyloxy, phenylcarbonyl, substituted phenylcarbonyl, phenylcarbonyloxy, substituted phenylcarbonyloxy, phenylcarbonylamino, substituted phenylcarbonylamino, phenoxy or substituted phenoxy;

R⁵ represents a straight or branched chain alkyl group containing up to six carbon atoms;

each Q independently represents C₁₋₄ alkyl or -CO₂R⁶ wherein R⁶ is C₁₋₄ alkyl;

m is zero, one or two;

n is zero or an integer from one to four;

r is one, two or three; and

p is zero or an integer from one to six

and any agriculturally acceptable metal chelate thereof.

Suitably, X is chloro, brome, nitro, cyano, C₁-C₄ alkyl, -CF₃, -S(O)-R¹ or -O

Suitably, X is chloro, bromo, nitro, cyano, C_1 - C_4 alkyl, - CF_3 , - $S(O)_mR^1$, or - OR^1 ; each Z is independently chloro, bromo, nitro, cyano, C_1 - C_4 alkyl, - CF_3 , - OR^1 , - $OS(O)_mR^5$ or - $S(O)_mR^5$; n is one or two; and p is zero, one or two.

Preferably, the 2-(substituted benzoyl)-1,3-cyclohexanedione of formula (I) is selected from the group consisting of 2-(2'-nitro-4'-methylsulphonylbenzoyl)-1,3-cyclohexanedione, 2-(2'-nitro-4'-methylsulphonyloxybenzoyl)-1,3-cyclohexanedione, 2-(2'-chloro-4'-methylsulphonylbenzoyl)-1,3-cyclohexanedione, 4,4-dimethyl-2-(4-methanesulphonyl-2-nitrobenzoyl)-1,3-cyclohexanedione, 2-(2-chloro-3-ethoxy-4-methanesulphonylbenzoyl)-5-methyl-1,3-cyclohexanedione and 2-(2-chloro-3-ethoxy-4-ethanesulphonylbenzoyl)-5-methyl-1,3-cyclohexanedione; most preferably is 2-(2'-nitro-4'-methylsulphonyl benzoyl)-1,3-cyclohexanedione.

The 2-(substituted benzoyl)-1,3-cyclohexanedione of formula (I) may exist in enolic tautomeric forms that may give rise to geometric isomers. Furthermore, in certain cases, the various substituents may contribute to optical isomerism and/or stereoisomerism. All such tautomeric forms, racemic mixtures and isomers are included within the scope of the present invention.

Agriculturally acceptable metal chelates of compounds of formula (I) are described in more detail in EP 0800317. In particular, metal ions which may be useful in forming the metal chelate compounds include di- and tri-valent transition metal ions such as Cu²⁺, Zn²⁺, Co²⁺, Fe²⁺, Ni²⁺ and Fe³⁺. The selection of a particular metal ion to form

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the metal chelate compound will depend upon the dione compound to be chelated. Those skilled in the art will readily be able to determine the appropriate metal ion for use with a specific dione compound, without undue experimentation. The preferred metal ions are divalent metal ions, particularly Cu²⁺, Zn²⁺, Co²⁺, with Cu²⁺ being especially preferred.

Suitably, the acetamide for use in the present invention is a chloroacetamide or an oxyacetamide.

In one embodiment of the invention, the acetamide is a chloroacetamide of formula (II)

$$\begin{array}{c|c} R^7 & R^9 \\ \hline \\ R^8 & O & CI \end{array} \hspace{1cm} \text{(II)}$$

wherein R⁷ is hydrogen, methyl or ethyl; R⁸ is hydrogen, methyl or ethyl; R⁹ is hydrogen or methyl; R¹⁰ is methyl, -OCH₃, -CH₂OCH₃, -OCH₂CH₃, -CH₂OCH₂CH₂CH₂CH₃, -OCH(CH₃)₂, -OCH₂CH₂CH₂CH₃ or a group

and A is S or CH=CH.

In one preferred embodiment, A is CH=CH; R⁷ is hydrogen, methyl or ethyl; R⁸ is hydrogen, methyl or ethyl; R⁹ is hydrogen or methyl; R¹⁰ is methyl, -OCH₃, -CH₂OCH₃, -CH₂OCH₂CH₂CH₃, -OCH(CH₃)₂, or -OCH₂CH₂CH₂CH₃.

Preferably, the chloroacetamide is selected from the group consisting of metolachlor, acetochlor and alachlor, preferably metolachlor, and most preferably, s-metolachlor.

In another preferred embodiment A is S; R^7 , R^8 and R^9 are methyl; and R^{10} is methoxymethyl. Preferably, the chloroacetamide is dimethenamide or p-dimethenamide.

In a further embodiment of the invention, the acetamide is an oxyacetamide of formula (III)

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wherein R^{11} is hydrogen, methyl, ethyl, propyl or isopropyl; R^{12} is hydrogen or halo; and R^{13} is a group

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Suitably, R¹¹ is methyl or isopropyl; R¹² is hydrogen or fluoro.

Preferably, the oxyacetamide is flufenacet or mefanacet; most preferably, flufenacet.

The glyphosate for use in the invention may be present as the free acid or as a suitable agriculturally acceptable salt. Such salts include, but are not limited to, the potassium, ammonium, isopropylammonium, sodium and trimethylsulfonium salts.

In one particularly preferred embodiment of the invention, the herbicidal combination for use in the method of the invention comprises 2-(2'-nitro-4'-methylsulphonylbenzoyl)-1,3-cyclohexanedione, glyphosate and s-metolachlor.

The rate at which the herbicidal components are applied will depend upon the particular type of weed to be controlled, the degree of control required, and the timing and method of application. In general, the components can be applied at an application rate of between about 400 g a.i./hectare (g/ha) and about 7750 g a.i./ha, based on the total amount of active ingredient. An application rate of between about 940 g a.i./ha and 3750 g a.i./ha is preferred. Suitably, the cyclohexanedione is applied at a rate of 20-300 g a.i./ha, preferably 40-250 g a.i./ha; the glyphosate compound is applied at a rate of 200-1400 g a.i./ha, preferably 400-1000 g a.i./ha; and the acetamide is applied at a rate of 200-6000 g a.i./ha, preferably 500-2500 g a.i./ha. In an especially preferred embodiment of this invention, the components are administered in relative amounts sufficient to provide an application rate of at least 2000 g a.i./ha, of which the cyclohexanedione provides at least 100 g/ha.

A still further aspect of the invention provides a method for the season-long control of unwanted vegetation as hereinbefore described, wherein said herbicidal combination further comprises one or more additional active ingredients. The additional active ingredient is suitably a pesticide, such as a herbicide, a fungicide, an insecticide, a nematocide or the like; preferably, the additional active ingredient is a herbicide. Examples of suitable herbicides include a triazine, such as atrazine, terbuthylazine, simazine etc. Further examples of herbicides which may be of use in the invention will be known to those skilled in the art.

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The components used in the method of the invention can be applied in a variety of ways known to those skilled in the art, at various concentrations. The combination of the invention is useful in controlling the growth of undesirable vegetation by pre-emergence or post-emergence application to the locus where control is desired. The combination of the invention is particularly effective when applied post-emergence, preferably early post-emergence.

The combinations of the invention can be used over a wide range of crops, such as corn (maize), wheat, rice, potato or sugarbeet. Suitable crops include those which are tolerant to one or more of the components in the composition. The tolerance may be natural tolerance produced by selective breeding or can be artificially introduced by genetic modification of the crop. Tolerance means a reduced susceptibility to damage caused by a particular herbicide compared to the conventional crop breeds. Crops can be modified or bred so as to be tolerant, for example to HPPD inhibitors like mesotrione, or EPSPS inhibitors like glyphosate. Corn (maize) is inherently tolerant to mesotrione.

The components used in the method of the invention may be administered simultaneously or sequentially. If administered sequentially, the components may be administered in any order in a suitable timescale, for example with no longer than 24 hours between the time of administering the first component and the time of administering the last component. Suitably, all the components are administered within a timescale of a few hours, such as one hour.

If the components are administered simultaneously, they may be administered separately or as a tank mix or as a pre-formulated mixture of all the components or as a pre-formulated mixture of some of the components tank mixed with the remaining components.

Therefore, a yet further aspect of the invention provides a herbicidal composition comprising a 2-(substituted benzoyl)-1,3-cyclohexanedione or metal chelate thereof, glyphosate or a salt thereof and an acetamide, provided that (i) when the 2-(substituted benzoyl)-1,3-cyclohexanedione is mesotrione, then the acetamide is not metolachlor, acetochlor, alachlor or dimethenamide, and (ii) when the acetamide is dimethenamide, then the 2-(substituted benzoyl)-1,3-cyclohexanedione is not 2-(2-chloro-4-methanesulfonylbenzoyl)-1,3-cyclohexanedione or 2-(4-methylsulfonyloxy-2-nitrobenzoyl)-4,4,6,6-tetramethyl-1,3-cyclohexanedione.

The compositions of the invention are useful as herbicides, demonstrating broadspectrum, season-long control of the unwanted vegetation. The composition can be used

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over a wide range of crops, such as corn (maize), wheat, rice, potato or sugarbeet. Suitable crops include those which are tolerant to one or more of the components in the composition. The tolerance may be natural tolerance produced by selective breeding or can be artificially introduced by genetic modification of the crop. Tolerance means a reduced susceptibility to damage caused by a particular herbicide compared to the conventional crop breeds. Crops can be modified or bred so as to be tolerant, for example to HPPD inhibitors like mesotrione, or EPSPS inhibitors like glyphosate. Corn (maize) is inherently tolerant to mesotrione.

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The herbicidal compositions of this invention also preferably comprise an agriculturally acceptable carrier therefore. In practice, the composition is applied as a formulation containing the various adjuvants and carriers known to or used in the industry for facilitating dispersion. The choice of formulation and mode of application for any given compound may affect its activity, and selection will be made accordingly. For example, the herbicidal composition of this invention may be a dustable powder, gel, a wettable powder, a water dispersible granule, a water-dispersable or water-foaming tablet, a briquette, an emulsifiable concentrate, a microemulsifiable concentrate, an oilin-water emulsion, a water-in-oil emulsion, a dispersion in water, a dispersion in oil, a suspoemulsion, a soluble liquid (with either water or an organic solvent as the carrier), an impregnated polymer film, or other forms known in the art. These formulations may be suitable for direct application or may be suitable for dilution prior to application, said dilution being made either with water, liquid fertilizer, micronutrients, biological organisms, oil or solvent. The compositions are prepared by admixing the active ingredients with adjuvants including diluents, extenders, carriers, and conditioning agents to provide compositions in the form of finely-divided particulate solids, granules, pellets, solutions, dispersions or emulsions. Thus, it is believed that the active ingredients could be used with an adjuvant such as a finely-divided solid, a mineral oil, a liquid of organic origin, water, various surface active agents or any suitable combination of these.

The active may also be contained in very fine microcapsules in polymeric substances. Microcapsules typically contain the active material enclosed in an inert porous shell which allows escape of the enclosed material to the surrounds at controlled rates. Encapsulated droplets are typically about 0.1 to 500 microns in diameter. The enclosed material typically constitutes about 25 to 95% of the weight of the capsule. The active ingredient may be present as a monolithic solid, as finely dispersed solid particles in either a solid or a liquid, or it may be present as a solution in a suitable solvent. Shell

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membrane materials include natural and synthetic rubbers, cellulosic materials, styrene-butadiene copolymers, polyacrylonitriles, polyacrylates, polyesters, polyamides, polyureas, polyurethanes, other polymers familiar to one skilled in the art, chemically-modified polymers and starch xanthates. Alternative very fine microcapsules may be formed wherein the active ingredient is dispersed as finely divided particles within a matrix of solid material, but no shell wall surrounds the microcapsule.

Suitable agricultural adjuvants and carriers that are useful in preparing the compositions of the invention are well known to those skilled in the art.

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Liquid carriers that can be employed include water, toluene, xylene, petroleum naphtha, crop oil, acetone, methyl ethyl ketone, cyclohexanone, acetic anhydride, acetonitrile, acetophenone, amyl acetate, 2-butanone, chlorobenzene, cyclohexane, cyclohexanol, alkyl acetates, diacetonalcohol, 1,2-dichloropropane, diethanolamine, pdiethylbenzene, diethylene glycol, diethylene glycol abietate, diethylene glycol butyl ether, diethylene glycol ethyl ether, diethylene glycol methyl ether, N,N-dimethyl formamide, dimethyl sulfoxide, 1,4-dioxane, dipropylene glycol, dipropylene glycol methyl ether, dipropyleneglycol dibenzoate, diproxitol, alkyl pyrrolidinone, ethyl acetate, 2-ethyl hexanol, ethylene carbonate, 1,1,1-trichloroethane, 2-heptanone, alpha pinene, dlimonene, ethylene glycol, ethylene glycol butyl ether, ethylene glycol methyl ether, gamma-butyrolactone, glycerol, glycerol diacetate, glycerol monoacetate, glycerol triacetate, glycerol triacetate, hexadecane, hexylene glycol, isoamyl acetate, isobornyl acetate, isooctane, isophorone, isopropyl benzene, isopropyl myristate, lactic acid, laurylamine, mesityl oxide, methoxy-propanol, methyl isoamyl ketone, methyl isobutyl ketone, methyl laurate, methyl octanoate, methyl oleate, methylene chloride, m-xylene, n-hexane, n-octylamine, octadecanoic acid, octyl amine acetate, oleic acid, oleylamine, o-xylene, phenol, polyethylene glycol (PEG400), propionic acid, propylene glycol, propylene glycol monomethyl ether, propylene glycol mono-methyl ether, p-xylene, toluene, triethyl phosphate, triethylene glycol, xylene sulfonic acid, paraffin, mineral oil, trichloroethylene, perchloroethylene, ethyl acetate, amyl acetate, butyl acetate, propylene glycol monomethyl ether and diethylene glycol monomethyl ether, methanol, ethanol, isopropanol, and higher molecular weight alcoholds such as amyl alcohol, tetrahydrofurfuryl alcohol, hexanol, octanol, etc., ethylene glycol, propylene glycol, glycerine, N-methyl-2-pyrrolidinone, and the like. Water is generally the carrier of choice for the dilution of concentrates.

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Suitable solid carriers include talc, titanium dioxide, pyrophyllite clay, silica, attapulgite clay, kieselguhr, chalk, diatomaceous earth, lime, calcium carbonate, bentonite clay, Fuller's earth, cotton seed hulls, wheat flour, soybean flour, pumice, wood flour, walnut shell flour, lignin, and the like.

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A broad range of surface-active agents are advantageously employed in both solid and liquid compositions, especially those designed to be diluted with carrier before application. The surface-active agents can be anionic, cationic, nonionic or polymeric in character and can be employed as emulsifying agents, wetting agents, suspending agents, or for other purposes. Typical surface active agents include salts of alkyl sulfates, such as diethanolammonium lauryl sulfate; alkylarylsulfonate salts, such as calcium dodecylbenzenesulfonate; alkylphenol-alkylene oxide addition products, such as nonylphenol-C.sub.18 ethoxylate; alcohol-alkylene oxide addition products, such as tridecyl alcohol-C.sub.16 ethoxylate; soaps, such as sodium stearate; alkylnaphthalenesulfonate salts, such as sodium dibutylnaphthalenesulfonate; dialkyl esters of sulfosuccinate salts, such as sodium di(2-ethylhexyl) sulfosuccinate; sorbitol esters, such as sorbitol oleate; quaternary amines, such as lauryl trimethylammonium chloride; polyethylene glycol esters of fatty acids, such as polyethylene glycol stearate; block copolymers of ethylene oxide and propylene oxide; and salts of mono and dialkyl phosphate esters.

Other adjuvants commonly utilized in agricultural compositions include crystallization inhibitors, viscosity modifiers, suspending agents, spray droplet modifiers, pigments, antioxidants, foaming agents, light-blocking agents, compatibilizing agents, antifoam agents, sequestering agents, neutralizing agents and buffers, corrosion inhibitors, dyes, odorants, spreading agents, penetration aids, micronutrients, emolients, lubricants, sticking agents, dispersing agents, thickening agents, freezing point depressants, antimicrobial agents, and the like. The compositions can also contain other compatible components, for example, other herbicides, herbicide safeners, plant growth regulants, fungicides, insecticides, and the like and can be formulated with liquid fertilizers or solid, particulate fertilizer carriers such as ammonium nitrate, urea and the like.

The invention will now be described in more details with reference to the following examples.

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A number of trial were carried out on a variety of weeds in a crop of maize using the following compositions:

1. Mesotrione/glyphosate

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- 2. Mesotrione/glyphosate/S-metolachlor
- 3. Mesotrione/glyphosate/S-metolachlor/atrazine

The glyphosate was administered at two different levels. Treatment was given early post-emergence of the crop. The level of weed control was assessed at various days after application (DAA). An improvement in weed control is seen with the three- and four-way combination over the two-way combination. The results are given in Table 1.

		g a.i./ha						
Mesotrione		105	105	105	105	105	105	
Glyphosate		560	560	560	841	841	841	
S-metolachlor			1051	1051		1051	1051	
Atrazine				392			392	
,	DAA		<u></u>	-				
Abutilon Theophrasti	29	99	99	99	99	99	99	
Abutilon Theophrasti	47	67	100	93	93	97	99	
Abutilon Theophrasti	34	99	99	99	99	99	99	
Amaranthus Retroflexus	47	100	100	100	100	100	100	
Amaranthus Retroflexus	41	99	99	99	99	99	99	
Amaranthus Retroflexus	42	100	100	100	100	100	100	
Amaranthus Rudis	47	100	100	100	100	100	100	
Amaranthus Rudis	47	100	100	100	98	100	100	
Amaranthus Rudis	42	97	93	97	97	92	98	
Amaranthus Tuberculatus	50	100	98	100	100	98	99	
Amaranthus Tuberculatus	38	96	100	100	97	99	99	
Ambrosia Artemisiifolia	39	78	83	89	72	80	98	
Ambrosia Artemisiifolia	57	74	85	95	78	93	95	
Ambrosia Artemisiifolia	45	95	93	98	97	95	98	
Ambrosia Artemisiifolia	38	100	100	100	98	98	100	
Ambrosia Trifida	57	85	96	92	92	98	92	

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Brachiaria Platyphylla	41	98	99	100	99	100	100
Chenopodium Album	39	94	93	97	95	96	98
Chenopodium Album	29	96	97.	97	92	97	98
Chenopodium Album	45	99	100	100	100	100	99
Chenopodium Album	29	98	99	99	96	99	99
Chenopodium Album	50 .	97	100	100	100	100	100
Chenopodium Album	47	93	100	100	96	96	96
Chenopodium Album	34	99	99	99	99	99	99
Chenopodium Album	46	100	100	100	100	100	100
Chenopodium Album	57	93	96	96	93	99	99
Convolvulus Arvensis	29	95	98	96	94	96	99
Digitaria ciliaris	42	77	97	97	94	97	96
Digitaria Sanguinalis	29	91	99	98	95	99	99
Digitaria Sanguinalis	47	75	82	87	80	82	83
Digitaria Sanguinalis	41	. 98	100	100	99	100	100
Echinochloa Crus-galli	45	97	100	100	98	100	100
Elusine Indica	28	95	92	97	100	95	95
Erichloa Villosa	47	45	83	92	65	83	88
Ipomoea Hederacea	38	95	95	88	97	77	87
Ipomoea Purpurea	50	92	95	98	92	98	97
Ipomoea Sp.	29	27	37	30	50	68	57
Ipomoea Sp.	41	90	90	92	92	93	90
Mollugo Verticillata	29	86	96	97	91	96	97
Panicum Dichotomiflorum	38	92	99	100	87	98	99
Raphanus Raphanistrum	39	96	95	97	96	96	98
Setaria Faberi	39	77	88	94	70	82	98
Setaria Faberi	57	90	95	98	97	97	95
Setaria Faberi	50	100	100	100	100	100	100
Setaria Faberi	34	93	98	98	96	98	98
Setaria Faberi	57	80	96	98	82	98	99
Setaria Glauca	41	85	93	96	91	96	97
Setaria Viridis	47	87	87	90	90	90	88

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Setaria Viridis	42	87	97	93	94	97	98
Sida Spinosa	41	99	99	100	100	100	99
Solanum Carolinense	29	13	46	52	0	53	73
Solanum Ptycanthum	34	99	99	99	99	99	99
Overall Mean		88	94	95	91	95	96